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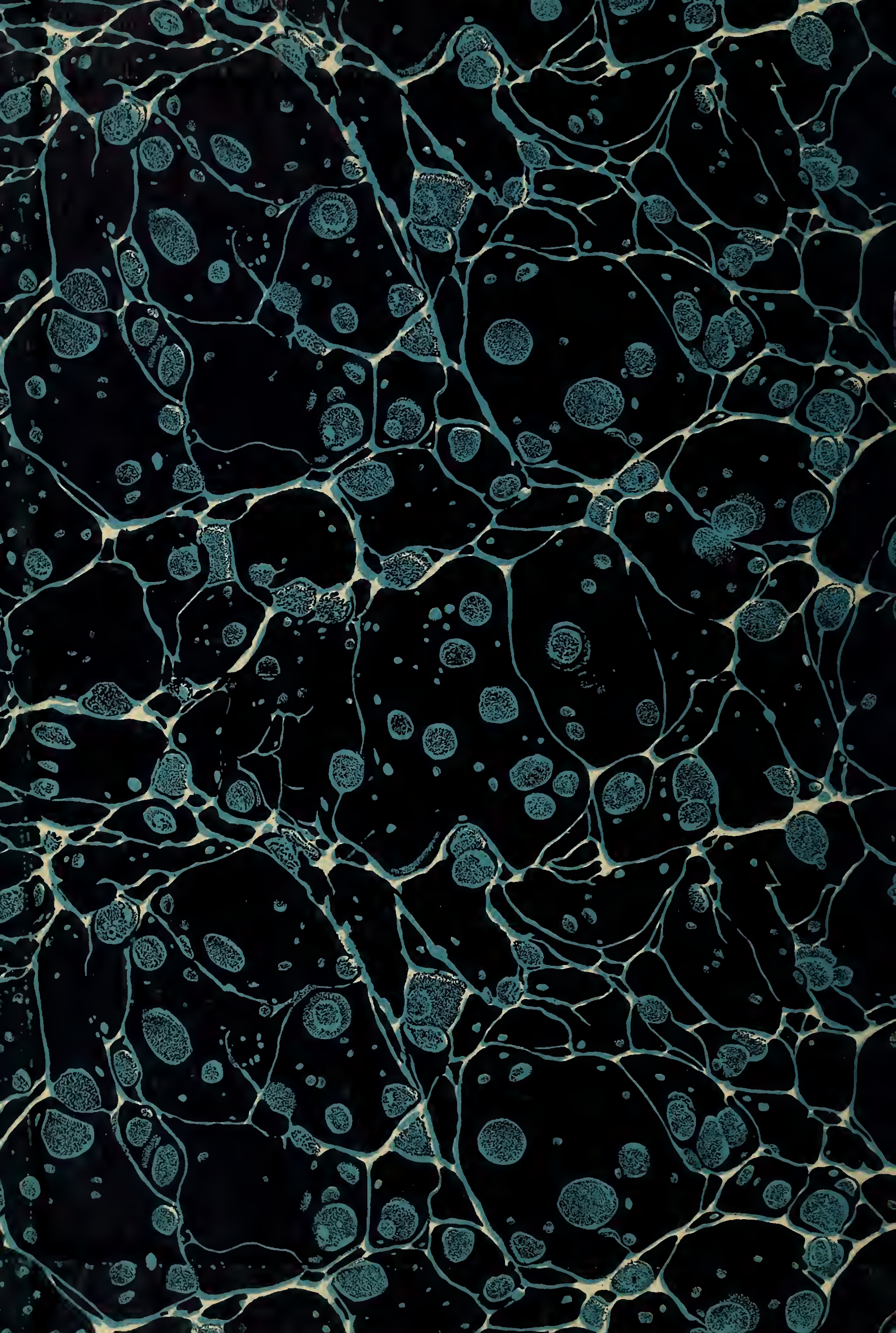

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DIRECT DETERMINATION OF INDIA RUBBER BY THE NITROSITE METHOD

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I. INTRODUCTION

One of the most important determinations in the analysis of a rubber compound is that of the amount of rubber present. The two types of methods used for this purpose are the direct and indirect.

The indirect methods comprise those in which the rubber is removed from the mineral fillers, either by ignition or solution. In such methods, it is usual to determine all the other constituents of the compound, and to call the remainder "rubber by difference." One method, which is very near the border line between these types, eliminates all volatile constituents except the rubber, and determines the latter by loss on ignition. In this instance, certain corrections are necessary to secure accurate results. While some of the indirect methods have considerable value, all of them are open to more or less objection.

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[1] Formerly associate chemist, Bureau of Standards, where all of the work described in this paper was done.

The direct methods include those in which definite compounds of rubber are formed. These are either weighed directly, or some important constituent is determined, and the rubber calculated by means of the proper factor. It is with a modification of one of these direct methods, viz, the nitrosite method, that this paper deals.

II. REVIEW OF PREVIOUS WORK ON NITROSITE METHOD

The original nitrosite method consists essentially in weighing the compound formed by treating rubber with the oxides of nitrogen which are evolved when nitric acid reacts with arsenic trioxide, starch, or some other reducing agents. The method was first proposed by Harries,[2] who has since published a number of articles on the subject. While other investigators have also done considerable work [3] it is not our purpose to discuss the various modifications proposed, other than to say it is generally accepted that the difficulty in securing a compound of definite composition renders the method of questionable value.

The work of Harries and others showed that there is no loss of carbon during the formation of the nitrosite. This led L. G. Wesson to the idea of burning the nitrosite, collecting the resulting carbon dioxide, and calculating from this the amount of rubber $(C_{10}H_{16})x$. The method as worked out by Wesson [4] was not entirely satisfactory because it could be used solely on soft vulcanized compounds containing only new rubber of good quality, and even then at times the results were very disappointing. All attempts to apply the method to compounds containing reclaimed rubber or substitutes failed. In view of the growing importance of the determination of rubber, and the difficulty in obtaining satisfactory results by means of the indirect methods, the authors of this paper took up the subject in order to ascertain whether or not a suitable method could be developed. A preliminary note of the work was presented orally to the rubber section of the American Chemical Society at its meeting in New York in September, 1916. At the same meeting Wesson presented a modification of his first method.[5] He believed that the difficulties with it could be eliminated by means of the wet combustion. In this modification, he worked with compounds containing only new rubber, mineral fillers, and sulphur, and while the results obtained were satisfactory, the method was still not of general application. The new method has been developed to the point where it is possible to secure excellent results on practically all classes of compounds.

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[2] Ber., 34 (1901), p. 2991; 35 (1902), p. 4429; 36 (1903), p. 1937; Z. Angew. Chem., 20 (1907), p. 1355 (1969).

[3] Alexander, Ber., 38 (1905), p. 181; 40 (1907), p. 1070; Z. Angew. Chem., 20 (1907), p. 1358; 24 (1911), p. 680; Gottlob, Z. Angew. Chem., 20 (1907), p. 2213. K. Fendler, Gummi Ztg., 24 (1910), p. 1000.

[4] L. G. Wesson, Bureau of Standards Technologic Paper No. 35; J. Ind. Eng. Chem., 6, p. 459; 1914.

[5] Wesson and Knorr, J. Ind. Eng. Chem., 9, p. 139; 1917.

III. ANALYTICAL PROCEDURE

Wesson's original article gives very full details of the method which he employed, and inasmuch as in our preliminary work we largely followed his method, it will be worth while to give a brief outline of it. The rubber is ground to pass a 20-mesh sieve. A sample of 0.25 to 0.50 g is taken for each determination, subjected to extraction with acetone, and after removal of most of the acetone, the sample is transferred to a 50 cm^3 graduated flask, and allowed to swell in chloroform. The nitrosite is now formed by passing through the chloroform solution a stream of oxides of nitrogen generated by heating arsenic trioxide and nitric acid. After standing overnight, the chloroform is decanted through a Gooch crucible and the residue is washed by decantation with chloroform. After driving off the last traces of the solvent by means of a gentle current of air, the nitrosite in the crucible is dissolved in acetone and the solution is returned to the original flask and made up to the mark. The flask is stoppered and shaken, or centrifuged, to throw down the mineral matter. A 25 cm^3 portion of the liquid is pipetted into another flask, most of the acetone removed by evaporation, and the nitrosite transferred to the combustion boat, the final rinsing being done with ethyl acetate. The nitrosite is dried in an air bath at 85° C for two hours, burned in the combustion furnace, and the carbon dioxide collected and weighed, and from it the rubber content is calculated.

It will be seen that this gives only the percentage of rubber which is originally insoluble in acetone. In order to obtain the total amount of rubber, it will be necessary to add to the rubber thus determined, the rubber resins found in the acetone extract. The sum should correspond to the percentage of rubber added to the compound.

IV. PRELIMINARY WORK

Our procedure in developing a satisfactory method was to check up each step of the one proposed by Wesson, in order to find out where errors occurred, and then to work out a satisfactory scheme for eliminating these errors.

1. COMBUSTION APPARATUS

During the combustion, the following products may be obtained which would require elimination or selective absorption in the absorption train: (1) Foreign matter in the oxygen, such as organic matter, moisture, etc.; (2) water; (3) oxides of nitrogen; (4) carbon dioxide from the nitrosite. The effects of these on the final results of our tests were ascertained by means of suitable blanks.

1. A blank determination on the oxygen was made under conditions identical with those of an actual determination. The train, arranged as used by Wesson, was as follows: Tower containing moist soda lime, bubble counter containing concentrated sulphuric acid, combustion tube, two U tubes containing glass beads and a solution of potassium bichromate in concentrated sulphuric acid, U tube with 30-mesh zinc, a special absorption tube containing soda lime followed by calcium chloride, a special absorption tube containing soda lime followed by alumina,[6] a U tube containing palladium-chloride solution. Wesson used alumina as the final drying agent, because of the claim that it is as efficient as sulphuric acid. It may be possible to prepare alumina having the same drying properties as sulphuric acid, but as a matter of general practice, the same material should be used before and after the soda-lime tubes.

Our first blanks showed a loss in weight of the soda-lime tubes, apparently because the alumina was less efficient than the concentrated sulphuric acid. We decided to avoid any uncertainty on this point by replacing the alumina with calcium chloride, and adding two new tubes containing bichromate and concentrated sulphuric acid, one just before the tube containing the zinc, and the other after the second soda-lime tube.[7] The latter was weighed after each determination. These tubes were filled at the same time with the same solution, so that we could be reasonably certain that the oxygen passing through the final tube which was weighed would have the same moisture content as it had when it entered the first. A new blank on the oxygen gave negative results.

2. To learn whether or not any water would be carried over into the soda-lime tubes, a small quantity of $BaCl_2.2H_2O$, containing about the quantity of water that would be formed during the combustion of the nitrosite, was heated in the boat in the usual manner.

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[6] Johnson, J., Am. Chem. Soc., 34, p. 911; 1912.

[7] The order of tubes adopted by Wesson is illustrated on p. 8 in the Bureau of Standards Technical Paper No. 35, while the authors' arrangement will be described in detail later on in this paper.

There was no appreciable change in weight, showing that no water vapor was carried over.

3. Copper nitrate was heated in the apparatus, to determine the effect of the oxides of nitrogen. No appreciable change in weight could be noted.

4. The ability of the apparatus to yield correct results for carbon dioxide was tested by burning pure sugar. The results of this experiment were satisfactory, and showed that the combustion furnace as arranged was adequate for the purpose.

2. NITRATION APPARATUS

When blanks on the nitration apparatus were made by conducting the nitrogen oxides through chloroform, a slight turbidity was noticed. The chloroform was evaporated off, and the residue soluble in acetone was transferred to a boat and burned in the usual manner. The amount of carbon dioxide thus obtained was considerable, amounting to 0.6 per cent or more, calculated as rubber. It was noticed that the rubber connections showed signs of having been attacked, and it was thought that small particles of rubber had been carried over mechanically. The rubber connections were then eliminated by sealing together the glass tubing. The one place where it was necessary to break the connection after each treatment was provided with a mercury seal. New blanks showed only 0.2 per cent. It is possible that by suitable purification of the chloroform, even this small amount could be eliminated. The chloroform which we used was of chemically pure quality, and was free from nonvolatile residue.

3. THE METHOD OF PROCEDURE

The nitrosite method is based upon the assumption that the carbon of the pure rubber substance is carried intact through the various steps in the determination, and is finally collected and weighed as carbon dioxide, and that carbon in other form which may be present in the rubber compound is eliminated at one step or another. During the nitration, mineral carbonates are decomposed with the elimination of their carbon dioxide. No organic fillers are known which upon nitration yield compounds insoluble in chloroform and soluble in acetone. If such were found, it would be, of course, necessary to devise some means of getting rid of them before proceeding with the determination of the rubber. We therefore worked upon the assumption that if we used pure gum rubber bands, no carbon other than that from the pure rubber substance would affect the result.

4. REMOVAL OF ACETONE

The first point investigated was the loss during the decantation of the chloroform from the nitrosite. The chloroform was evaporated off, and the residue transferred to a boat and burned as usual. The carbon dioxide thus found was equal to that obtained from 4 to 8 per cent of rubber. In an effort to eliminate this error, acetone was substituted for the chloroform in the nitration. Considerable heat was evolved, which showed that some new reaction took place. It was then found that when oxides of nitrogen were passed into redistilled acetone a considerable amount of an oily product was formed. No attempts were made to identify these products of the reaction. Check determinations were now made in which in one case, the acetone was completely removed by drying in hydrogen at 85 to 90° C, and in the other the sample was treated as directed by Wesson. The results were significant. In the chloroform solution, the acetone-free sample showed a residue of only 0.7 per cent, calculated as rubber, while the samples containing acetone showed over 9 per cent. Thus while the organic matter in the chloroform filtrate may largely result from the reaction between the oxides of nitrogen and acetone left in the rubber, there is no doubt that there is still an appreciable amount which can not be accounted for in this way. Under the circumstances, it is only reasonable to conclude that this organic matter was originally present in the rubber itself. The nature of this latter material which is carried over in the chloroform is, of course, not known, and we have had no opportunity to investigate it. The results given by Wesson in his original paper showed that a sample after standing for some weeks gave results from 1 to 2 per cent lower than those obtained when the sample was fresh. The change in the sample was probably due to oxidation, and it may be that the oxidized material does not follow the same course as the unchanged rubber. If so, this would explain why a small amount remains in the chloroform, since it is well known that even fresh rubber contains a small percentage of oxidized rubber.

It is apparent that the organic matter remaining in the chloroform from the samples which have been freed from acetone is part of the rubber. We therefore recover this portion of the rubber and include it with the nitrosite insoluble in chloroform. After filtering off the nitrosite, the filtrate is evaporated to dryness and that portion of the residue which is soluble in acetone is added to the solution in acetone of the main portion of the nitrosite.

Wesson transferred his acetone solution of the nitrosite to a 50 cm^3 flask from which he eventually took 25 cm^3 for analysis. Thus each nitration yielded only one determination. We have replaced the graduated flask with a weight burette of a special form. (See Fig. 1.) The small cup in the bottom collects the mineral sediment, allowing the clear liquid to be drawn off through the stopcock. The burette is, of course, weighed before and after drawing off the liquid. The weight of the burette empty is known, and consequently the total amount of liquid and the portion drawn off for analysis can easily be calculated. There is an advantage in using the weight burette in that other portions can be drawn off for check determinations.

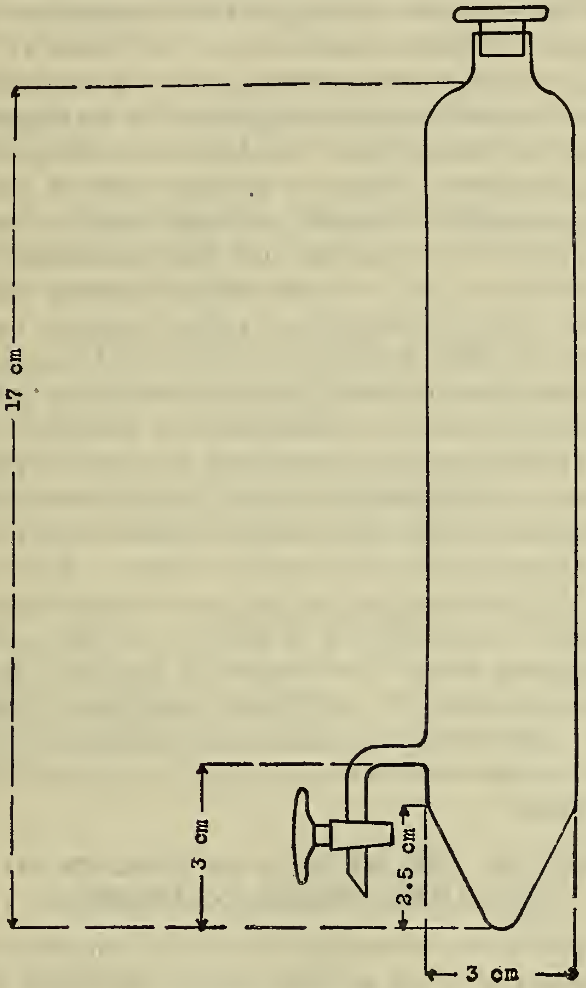

Fig. 1. *Weight burette*

5. DRYING

There was one more point where an error of considerable size could be introduced unless uniform conditions were maintained. This was in the drying of the nitrosite previous to combustion. Wesson recommended drying at 85° C for 2 hours. A number of determinations were made in which the further loss in weight after the first 2 hours' drying at this temperature was noted. These results varied from 0.007 to 0.017 g. About 15 hours were required to obtain constant weight. Some of this loss in weight may have been moisture originally present in the organic solvents, and, of course, this moisture would have no effect on the determination of carbon. However, at least part of this loss undoubtedly represented organic solvents held by the gummy nitrosite, and to this extent an error was introduced. It is quite possible, therefore, that some of Wesson's results which agreed very closely with the amount of rubber actually put into the compound were really obtained by a fortunate balancing of errors.

It is evident that 15 hours' drying is out of the question. In Wesson's work on the wet combustion he attempted to remedy this defect, which we had pointed out to him in a private communication. After drying for a short time to remove the greater part of the acetone and ethyl acetate, he added water and hydrochloric acid, and again evaporated to dryness. The objection to the use of hydrochloric acid is that the nitrosite is insoluble in acid solution; consequently it is difficult for the water to penetrate the gummy mass. The nitrosite is soluble in alkalies, and we therefore moistened it with water made faintly alkaline with ammonia. The results were exceedingly satisfactory; in this way we found we were able to remove all of the organic solvent in a very short time.

V. DETAILS OF THE METHOD AS APPLIED TO VARIOUS VULCANIZED RUBBER COMPOUNDS

By the preliminary work described above a method was developed by means of which we were able to determine with a considerable degree of accuracy the percentage of rubber present in compounds containing only new rubber, sulphur, and inorganic mineral fillers. Our efforts were then directed toward extending the use of the method to such compounds as contained reclaimed rubber, lampblack, bituminous substances or the so-called "mineral rubbers," oil substitutes, etc.

1. COMPOUNDS CONTAINING RECLAIMED RUBBER

No changes were found to be necessary in order to apply the method to compounds containing reclaimed rubber. In such cases, of course, the acetone extract is much higher than usual, owing to the presence of mineral oils in the reclaimed rubber, and it would not be just to add the total amount of the acetone extract to the rubber hydrocarbons determined, in order to ascertain the actual amount of rubber added. An arbitrary correction must be made, and it probably will be sufficiently accurate if it is assumed that the acetone extract of the rubber itself is 4 per cent, as indicated by the experience of this Bureau. The rubber hydrocarbons determined are, therefore, 96 per cent of the total rubber present, and the proper calculations should be made. The corrected figure thus obtained will probably be more accurate than that obtained by any other method in use to-day.

2. COMPOUNDS CONTAINING LAMPBLACK

Lampblack offered no special difficulties in the way of determining the amount of rubber present. It was not acted upon during nitration, and while some of it was carried over with the nitrosite into the acetone solution, it was completely separated from the latter when it collected in the cup in the bottom of the weight burette.

3. COMPOUNDS CONTAINING BITUMINOUS SUBSTANCES

A slight modification of the ordinary method was found necessary whenever bituminous substances were present. The sample was extracted with acetone as usual. If after drying, upon placing the sample in the chloroform, a dark-colored solution resulted, the solution was filtered through a paper extraction thimble, the sample and filtrate were placed in the extraction apparatus, and the sample was extracted with chloroform for four hours. After extraction the rubber was transferred to the nitration apparatus, fresh chloroform added, and the nitration carried on as usual. The rest of the method was as before.

The calculation of the percentage of rubber from these tests is a more complicated problem. The acetone and chloroform extracts contain, in addition to the soluble portions of the mineral rubber, certain resins and other substances of the rubber. It is therefore necessary to assume an arbitrary correction, based upon the amount of material ordinarily extracted by these reagents from new rubber. We have already stated that we have taken 4 per cent as a fair value for acetone-soluble constituents. The chloroform extract of correctly vulcanized rubber is less than this, and we have arbitrarily chosen 2 per cent. This latter figure was derived from the results of a large number of tests made at this Bureau over a number of years. If reclaimed rubber is present this figure will be low, but even under such conditions we believe the differences will not be such as to materially lessen the value of the determination.

4. COMPOUNDS CONTAINING OIL SUBSTITUTES

Tests made on compounds containing oil substitutes gave very high results for rubber, showing that part at least of the oil substitutes was carried along in the same manner as the nitrosite. It is evident, therefore, that these substitutes must be removed before nitration. In the course of routine analyses, the rubber is extracted with acetone, and, in the absence of mineral rubbers, is then treated with alcoholic potassium or sodium hydroxide.[8] The same procedure was followed in this work, and after extraction with alkali the sample was dried, suspended in chloroform, and nitrated in the usual way.

VI. DISCUSSION OF RESULTS

TABLE 1.—Composition of Samples Tested

	Sample number				
	E159	BS45	BS53	BS65	BS109
Rubber:	Per cent	Per cent	Per cent	Per cent	Per cent
Smoked sheets	33				
Pale crepe		30	48	49.2	9.1
Reclaimed					[a] 79.8
Substitutes:					
Black corn No. 2		15			
M. R. X.				24.6	
Fillers:					
Lampblack			4		
Whiting	29	25			
Zinc oxide	30	20	22		
Litharge	5	7	24	24.6	6.4
Sulphur	2	3	2	1.6	4.7
Paraffin	1				

[a] This reclaimed rubber, on analysis, gave 61.3 per cent rubber and organic extract. The percentage of 79.8 reclaimed corresponds to 48.9 per cent in the rubber from this source, which with the 9.1 per cent of new rubber gives a total rubber content of 58 per cent.

In Table 1, we give the composition of the samples used specially in this work. Sample E159 was prepared by a rubber company for another investigation, and the formula given is that furnished by it. From the results obtained in this other investigation, we believe the composition as given to be accurate. The other samples were mixed and vulcanized at this Bureau. Table 2 gives the results obtained on these samples when using our method.

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[8] See Bureau of Standards Circular No. 38, p. 71.

TABLE 2.—Results of Analyses

Sample No.	Rubber calculated per cent	Rubber found				
		Per cent	Per cent	Per cent	Per cent	Per cent
E159 [a]	33.0	34.2	32.6	33.5	34.8	33.1
BS45................................	30.0	29.8	30.0	30.6	30.6	....
BS53................................	48.0	48.7	48.6	48.1	....	....
BS65................................	49.2	49.4	49.2	48.9	48.9	....
BS109................................	58.0	Series a		56.6	57.2	58.8
		Series b		59.3	59.4	60.8
		Series c		55.9	56.6	56,7
		Series d		58.1	59.5	60.2
		Average of 12 determinations				58.3

[a] The results on E159 were obtained before adopting ammonia in the drying of the nitrosite before combustion. The somewhat higher results in two of the determinations are undoubtedly caused by the occlusion of small quantities of organic solvents.

From the foregoing it is apparent that with compounds containing only new rubber, a high degree of accuracy is attainable. Lampblack does not interfere with the analysis. When reclaimed rubber is present, there is apt to be a greater variation in the results than when only new rubber is present. This is not surprising, for it must be realized that with material of this nature there is bound to be considerable variation in composition in the material itself, and we must expect the results to vary more than usual. With further experience it may be found that slight changes in the manipulation will bring about increased accuracy, but even now we believe that the results are sufficiently accurate for all practical purposes.

It should be remembered that this method does not attempt to discriminate between new and reclaimed rubber; as far as we know there is no quantitative method to-day which will determine the percentage of either kind in a mixture of the two. Our method gives merely the total of the two kinds, and the average quality must be determined by means of the usual mechanical tests of tensile strength, elongation, permanent set, etc.

When oil substitutes are present, the results are again not as good as when only new rubber is used, but on the whole are satisfactory and usually more accurate than when reclaimed rubber is present.

We have as yet found no fillers, organic or inorganic, which interfere with the accuracy of the method. Mineral carbonates are decomposed in the acid solution and the carbon dioxide is driven off during evaporation. As will be seen from Table 1, we tested compounds containing from 25 to 28 per cent of whiting without having it affect the results in the least. Other carbonates would act in the same way. We have had occasional tests when filter paper was allowed to remain with the rubber during the nitration. It was eliminated during the filtration of the nitrosite from the chloroform solution, since it is insoluble in acetone, and the nitrosite is soluble. The only fillers which could possibly affect the analysis are those which are insoluble in acetone, chloroform, and alcoholic potassium hydroxide, and which upon nitration produce new compounds which are soluble in acetone. With a few preliminary tests, which are usually made, it is a very simple matter to change the procedure so as to eliminate mineral rubbers and oil substitutes before they can do any harm.

During the early part of this investigation we used the furnace described by Wesson. Later, we were unable to obtain a further supply of combustion tubing. We therefore had recourse to fused silica tubes, but these presented new problems which had to be solved. It was no longer possible to fuse platinum wires in the wall of the tube, or even to draw out the latter as Wesson had done. We therefore cemented a reducing tube of Pyrex glass to the silica tube, using de Khotinsky cement. The Pyrex reducing tube at one end was just large enough to slip over the silica tube, and the other end was drawn down to the same size as that of the glass tubing on the first absorption bottle. For a catalyzer we used platinized asbestos, heated with a gas flame. The radiation from this heating occasionally softened the de Khotinsky cement and we arranged a cooling device by wrapping the silica-Pyrex joint with a strip of cotton cloth about 4 cm wide, the loose end of which dipped down into a beaker of water. The cotton strip was thus kept moist and effectually kept the temperature of the cement at a safe point.

VII. METHOD FINALLY ADOPTED

Preliminary extractions of a 2 g sample are made according to the usual methods of the Bureau of Standards[9] to determine the amount of acetone, chloroform, and alcoholic potash extracts.

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[9] Bureau of Standards Circular No. 38.

These extractions will prove the presence or absence of mineral rubbers and oil substitutes. In the absence of both, proceed as below. If mineral rubbers are present, make a chloroform extraction; if oil substitutes, an alcoholic potash extraction; and if both are present make both extractions. In every case make these extractions after the acetone extraction, and before the sample is allowed to swell in chloroform.

Take 0.500 to 1.000 g of the finely-ground sample (call this weight *W*), extract with acetone for 8 hours, and if necessary with chloroform and alcoholic potash. Dry the residue in hydrogen (or other inert gas) for 2 hours at 100° C. Place the sample in 50 to 75 cm^3 of chloroform, and allow it to swell. Immerse the flask containing the rubber in cold water and pass into this, until the green color which is formed persists for 30 minutes, the gases formed by heating arsenic trioxide and nitric acid of specific gravity 1.30. It is important here, in order to avoid contamination, that no rubber connections be used. Allow the solution to stand overnight; the next day, filter off the nitrosite through a Gooch crucible and wash with small quantities of chloroform. Remove the acid gases and chloroform from the flask by means of a gentle current of air. Evaporate the filtrate to dryness. Dissolve in acetone the nitrosite remaining in the flask, on the Gooch crucible, and in the residue from the filtrate, and filter the solution through asbestos into the weight burette. The total volume should be about 100 cm^3. Allow this solution to stand a short time so as to permit any sediment which may form to settle out in the bottom of the weight burette. Weigh the burette before and after filling, calling the difference *N*. Draw off about 25 cm^3 into a small Erlenmeyer flask, reweigh the burette and call the difference *O*. Evaporate the portion drawn off to a small volume, transfer to a porcelain boat (about 14 cm long, and 1 cm wide) which has been filled with alundum, and wash the flask with acetone. It is best to make this transfer in small portions, drying the boat and contents for a few minutes between each addition. After the final washing and drying, add 1 or 2 cm^3 of a 1 per cent solution of ammonia in distilled water and dry in an inert gas for 1 hour at 90° C. Repeat with a second portion of ammonia and dry as before. By this means, all of the organic solvent will be removed.

Place the boat in the furnace and proceed with the combustion as usual. The furnace, of whatever type used, should be carefully tested before using. Pass the products of combustion through

U tubes or other satisfactory absorption tubes, placed in the following order; *a, b, c*, potassium bichromate-concentrated sulphuric acid; *d*, powdered zinc, 20-mesh; *e, f*, soda lime and calcium chloride; *g*, potassium bichromate-concentrated sulphuric acid; *h*, dilute palladium chloride solution. Very little palladium chloride is needed. We use about one drop of 10 per cent solution in 10 cm^3 of distilled water. Weigh *e, f*, and *g* before and after each combustion; refill *c* and *g* frequently from the same solution so that the gases which enter *e* and those that leave *g* will have the same moisture content. The palladium chloride serves to detect the presence of carbon monoxide; if there is any blackening, it shows incomplete oxidation. In this event, discard the results and repeat the determination.

The carbon dioxide will equal the algebraic sum of the differences in tubes *e, f*, and *g*. Call this *P*. The factor for calculating from carbon dioxide to rubber hydrocarbons is 0.309. The formula is therefore as follows:

$P \times 0.309 \times \frac{N}{O} \times \frac{100}{W} =$ Percentage of rubber hydrocarbons. Correct this figure for whatever extractions were made previous to nitration.

VIII. SUMMARY

Harries and his coworkers claimed that there is no loss of carbon when the rubber molecule passes through the process for the formation of the nitrosite. Wesson's combustion method for the direct determination of rubber depends upon the correctness of this claim. An examination of his method shows that, although he obtained fairly satisfactory results, there are certain avoidable sources of error. Compounds containing new rubber and no substitutes or organic fillers, can be analyzed with correct results, owing to a balancing of errors.

By suitable changes in the procedure of Wesson the present authors have succeeded in obtaining very satisfactory results, not only with pure gum compounds, but also with compounds made up with reclaimed rubber, mineral rubber, oil substitutes, lampblack, etc.

WASHINGTON, June 20, 1919.

⁂